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14. ABSTRACT Techniques were developed for producing epitaxial layers of ZnTe _{1-x} Se _x that are lattice-matched to GaSb substrates for use as buffer layers for Hg _{1-x} Cd _x Se IR-detection layers. Dislocation density values of 7 X 10 ⁴ cm ⁻² are obtained from confocal photoluminescence images of the ZnTe _{1-x} Se _x layers. Techniques were developed for producing epitaxial layers of Hg _{1-x} Cd _x Se with x-values appropriate for optical absorption in the MWIR and LWIR spectral regions. Background carrier concentration values as low as 8 x 10 ¹⁵ cm ⁻³ were achieved in LWIR with promising excess carrier lifetimes. IR Cathodoluminescence was demonstrated to 5.5 micrometer.					
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Report Title

Final Report - Synthesis and Characteristics of HgCdSe for IR Detection

ABSTRACT

Techniques were developed for producing epitaxial layers of $\text{ZnTe}_{1-x}\text{Se}_x$ that are lattice-matched to GaSb substrates for use as buffer layers for $\text{Hg}_{1-x}\text{Cd}_x\text{Se}$ IR-detection layers. Dislocation density values of $7 \times 10^4 \text{ cm}^{-2}$ are obtained from confocal photoluminescence images of the $\text{ZnTe}_{1-x}\text{Se}_x$ layers. Techniques were developed for producing epitaxial layers of $\text{Hg}_{1-x}\text{Cd}_x\text{Se}$ with x-values appropriate for optical absorption in the MWIR and LWIR spectral regions. Background carrier concentration values as low as $8 \times 10^{15} \text{ cm}^{-3}$ were achieved in LWIR with promising excess carrier lifetimes. IR Cathodoluminescence was demonstrated to 5.5 micrometer.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
03/11/2014 13.00	J. Chai, O. C. Noriega, A. Dedigama, J. J. Kim, A. A. Savage, K. Doyle, C. Smith, N. Chau, J. Pena, J. H. Dinan, D. J. Smith, T. H. Myers. Determination of Critical Thickness for Epitaxial ZnTe Layers Grown by Molecular Beam Epitaxy on (211)B and (100) GaSb Substrates, Journal of Electronic Materials, (06 2013): 0. doi: 10.1007/s11664-013-2650-8
08/25/2012 5.00	J. Chai, O. C. Noriega, J. H. Dinan, T. H. Myers. Critical Thickness of ZnTe on GaSb(211)B, Journal of Electronic Materials, (05 2012): 0. doi: 10.1007/s11664-012-2120-8
08/25/2012 3.00	Kyoung-Keun Lee, Kevin Doyle, Jessica Chai, John H. Dinan, Thomas H. Myers. X-Ray Photoelectron Spectroscopy Study of Oxide Removal Using Atomic Hydrogen for Large-Area II–VI Material Growth, Journal of Electronic Materials, (04 2012): 0. doi: 10.1007/s11664-012-2085-7
08/25/2012 4.00	J. Chai, K.-K. Lee, K. Doyle, J.H. Dinan, T.H. Myers. Growth of Lattice-Matched ZnTeSe Alloys on (100) and (211)B GaSb, Journal of Electronic Materials, (03 2012): 0. doi: 10.1007/s11664-012-2054-1
08/25/2013 6.00	Kevin Doyle, Craig H. Swartz, John H. Dinan, Thomas H. Myers, Gregory Brill, Yuanping Chen, Brenda L. VanMil, Priyalal Wijewarnasuriya. Mercury cadmium selenide for infrared detection, Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures, (05 2013): 124. doi: 10.1116/1.4798651
TOTAL:	5

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

“Electron Transport in HgCdSe”, Kevin Doyle, Gregory Brill, Craig Swartz, Thomas Myers, 2013 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Chicago IL (October 1-3, 2013)

“Exploring the Optical Properties of Hg_{1-x}Cd_xSe Films Using IR-Spectroscopic Ellipsometry”, Frank Peiris, Gregory Brill, Brenda VanMil, Kevin Doyle, Thomas Myers, 2013 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Chicago IL (October 1-3, 2013)

“The Use of Confocal Photoluminescence Microscopy for Determination of Defect Densities in Various II-VI Semiconductors”, O.C. Noriega, A. Savage, T.H. Myers, P.J. Smith, R.N. Jacobs, C.M. Lennon, P.S. Wijewarnasuriya, and Y. Chen, 2013 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Chicago IL (October 1-3, 2013)

“Use of Atomic Hydrogen to Prepare GaSb(211)B and GaSb(100) Substrates for Subsequent ZnTe Growth by MBE”, T.H. Myers, J. Chai, J.J. Kim, K.-K. Lee, O.C. Noriega, A. Savage, K. Doyle, D. J. Smith, J.H. Dinan, 40th Conference on the Physics and Chemistry of Surfaces and Interfaces, January 20-24, 2013, Waikoloa, Hawaii, USA

“Comparison of Cathodoluminescence, Imaging Micro-Photoluminescence and Confocal Photoluminescence Microscopy for Determination of Interfacial Defect Densities in Semiconductors”, O.C. Noriega, A. Savage, J. Chai, A. Dedigama, K. Doyle, N. Chau (ST), J. Pena (ST), J.J. Kim, D. J. Smith, J.H. Dinan, T.H. Myers, 40th Conference on the Physics and Chemistry of Surfaces and Interfaces, January 20-24, 2013, Waikoloa, Hawaii, USA

“Comparison of Cathodoluminescence, Imaging Micro-Photoluminescence and Confocal Photoluminescence Microscopy for Determination of Defect Densities in Semiconductors”, O.C. Noriega, A. Savage, J. Chai, A. Dedigama, K.-K. Lee, K. Doyle, C. Smith, N. Chau (ST), J. Pena (ST), J.J. Kim, D. J. Smith, J.H. Dinan, T.H. Myers, 2012 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Seattle, WA (November 2012)

“MBE growth of ZnTe and ZnTeSe on GaSb”, J. Chai, K.-K. Lee, K. Doyle, C. Smith, N. Chau (ST), J. Pena (ST), A. Dedigama, J.H. Dinan, O.C. Noriega, J.J. Kim, D. J. Smith, T.H. Myers, 2012 North American Molecular Beam Epitaxy Conference, Stone Mountain, GA (October 2012)

“Residual Doping in Hg_{1-x}Cd_xSe,” K. Doyle, C.H. Swartz, T.H. Myers, J. H. Dinan, G. Brill, Y. Chen, B. L. VanMil, P. Wijewarnasuriya, K. Lund, M. Weber, K. Lynn. 2012 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Seattle, WA (November 2012)

“Hg(1-x)Cd_xSe Growth for Focal Plane Arrays,” K. Doyle, G. Brill, Y. Chen, B.L. VanMil, C.H. Swartz, T. H. Myers and J. Dinan. 2012 North American Molecular Beam Epitaxy Conference, Stone Mountain, GA (October 2012)

"Overview of Hg_{1-x}Cd_xSe Material Research for IR Applications,” K. Doyle, G. Brill, Y. Chen, T. H. Myers, S. Trivedi. 2012 SPIE Conference, San Diego CA (August 2012)

“Determination of critical thickness for epitaxial ZnTe layers on (211)B and (100) GaSb substrates grown by molecular beam epitaxy” J. Chai, O.C. Noriega, K. Doyle, A. Dedigama, J.J. Kim, A.A. Savage, C. Smith, N. Chau, J. Pena (ST), J.H. Dinan, D. J. Smith, and T.H. Myers. International workshop on 6.1 Å II-VI and III-V materials and their integration, Tempe AZ (November 2011)

“Critical Thickness Study of ZnTe on GaSb(211)B”, Jessica Chai, John H. Dinan, Thomas H. Myers, . International workshop on 6.1 Å II-VI and III-V materials and their integration, Tempe AZ (November 2011)

“Growth of HgCdSe for LWIR Detection,” K. Doyle, G. Brill, J. Chai, K. Lee, J. Dinan, T.H. Myers. International workshop on 6.1 Å II-VI and III-V materials and their integration, Tempe AZ (November 2011)

“Optical and Electrical Properties of HgCdSe,” K. Doyle, G. Brill, J. Chai, K. Lee, J. Dinan, T.H. Myers. 2011 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Chicago IL (October, 2011)

"Xray photoelectron spectroscopy study of oxide removal using atomic hydrogen for large-area II-VI material growth," K. Lee, K. Doyle, J. Chai, W. Priyantha, J. Dinan, T. H. Myers. 2011 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Chicago IL (October, 2011)

“Growth of lattice-matched CdSeTe and ZnTeSe alloys on (100) and (211)B GaSb,” J. Chai, K. Lee, K. Doyle, W. Priyantha, J. Dinan, T. H. Myers. 2011 U.S. Workshop on the Physics and Chemistry of II-VI Materials, Chicago IL (October, 2011)

“Optical and Electrical Properties of HgCdSe,” K. Doyle, G. Brill, J. Chai, K. Lee, J. Dinan, T.H. Myers. 15th International Conference on II-VI Compounds, Mayan Riviera, Mexico (August 2011)

"Xray photoelectron spectroscopy study of oxide removal using atomic hydrogen for large-area II-VI material growth," K. Lee, K. Doyle, J. Chai, W. Priyantha, J. Dinan, T. H. Myers. 15th International Conference on II-VI Compounds, Mayan Riviera, Mexico (August 2011)

“Growth of lattice-matched CdSeTe and ZnTeSe alloys on (100) and (211)B GaSb,” J. Chai, K. Lee, K. Doyle, W. Priyantha, J. Dinan, T. H. Myers. 15th International Conference on II-VI Compounds, Mayan Riviera, Mexico (August 2011)

Preliminary Results for Atomic Hydrogen Cleaning of GaSb for II-VI Materials Growth, Kevin Doyle, Kyoung-Keun Lee, Weerasinghe Priyantha, John Dinan, Thomas H. Myers, The 2010 U.S. WORKSHOP on the PHYSICS and CHEMISTRY of II-VI MATERIALS, October 26–28, 2010, New Orleans, Louisiana

Invited Talks - Thomas H Myers

New Approaches for Characterization of Heterogeneous Material Integration Quality”, 2013 North American Molecular Beam Epitaxy Conference -Post-Conference Workshops,, Banff, Alberta, Canada (October 5-11 2013)

“MBE growth of ZnTe and ZnTeSe on GaSb”, Joint Department of Electrical Engineering and Department of Physics Colloquium, University at Buffalo, Buffalo, NY (Sept 2012).

Invited Panel Member, “Infrared Detectors and MBE: Made for each other?”, 2012 North American Molecular Beam Epitaxy Conference, Stone Mountain, GA (October 2012)

Molecular beam epitaxy of II-VI materials on GaSb, T.H Myers, International workshop on 6.1 Å II-VI and III-V materials and their integration, 11/8-9, 2011, Arizona State University, Tempe, AZ

Number of Presentations: 24.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

08/27/2013 7.00 K. Doyle, G. Brill, Y.Chen, T.H. Myers, C.H. Swartz. HgCdSe Material Research for IR Applications, Infrared Sensors, Devices, and Applications II. 15-AUG-12, . : ,

TOTAL: **1**

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

<u>Received</u>	<u>Paper</u>
03/11/2014 14.00	F. C. Peiris, G. Brill, Kevin J. Doyle, Brenda VanMil, Kevin J. Doyle, Thomas H. Myers. EXPLORING THE OPTICAL PROPERTIES OF Hg1-xCdxSe FILMS USING IR-SPECTROSCOPIC ELLIPSOMETRY, Journal of Electronic Materials (08 2013)
08/27/2013 11.00	J. Chai, O.C. Noriega, A. Dedigama, J.J. Kim, A.A. Savage, C. Smith, N. Chau, J. Pena, J.H. Dinan, D.J. Smith, T.H. Myers. Determination of Critical Thickness for Epitaxial ZnTe layers grown by molecular beam epitaxy on (211)B and (100) GaSb Substrates , Journal of Materials Research (10 2013)
TOTAL:	2

Number of Manuscripts:

Books

<u>Received</u>	<u>Paper</u>
TOTAL:	

Patents Submitted

Patents Awarded

Awards

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	<u>Discipline</u>
Kevin J Doyle	1.00	
FTE Equivalent:	1.00	
Total Number:	1	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	
Jessica Chai	0.10	
Craig Swartz	0.03	
FTE Equivalent:	0.13	
Total Number:	2	

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
John H Dinan	0.50	
Thomas H Myers (Cost Share)	0.08	
Terry Golding (Cost Share)	0.06	No
FTE Equivalent:	0.64	
Total Number:	3	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Joseph Pena	0.00	Physics
Nelson Chau	0.00	Physics
FTE Equivalent:	0.00	
Total Number:	2	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

<u>NAME</u>
Total Number:

Names of personnel receiving PHDs

<u>NAME</u>
Kevin J. Doyle
Total Number:

Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Alyssa A Savage	0.04
Odille C.N. Myers	0.17
FTE Equivalent:	0.21
Total Number:	2

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

During this program, we significantly advanced the state of the art in five areas.

- Molecular Beam Epitaxy of ZnTe layers on GaSb substrates
- Molecular Beam Epitaxy of ZnSeTe layers on GaSb substrates
- Molecular Beam Epitaxy of HgCdSe layers on composite Si/ZnTe substrates
- Luminescence imaging techniques for dislocation determination,
- Demonstrating cathodoluminescence out to 5.5 μm on various IR materials

Much of this work has been published. The HgCdSe work is the subject of a doctoral dissertation that has been accepted. For details, we refer the reader to the prior interim reports, the manuscripts and the dissertation that have uploaded as part of this and prior reports. In this section of the report, we summarize highlights of the accomplishments.

1. MBE of ZnTe

During Year Two, we had deposited layers of ZnTe with various thicknesses on (211)B GaSb substrates. This orientation was chosen in anticipation of the use of a GaSb/ZnSeTe composite substrate for MBE of HgCdSe. We interpreted the dramatic reduction in the FWHM value of Bragg x-ray reflections with thickness as an indication that relaxation of the lattice parameter took place for thicknesses in the range 350nm to 375 nm. We associated this value with the critical thickness value h_c that is a characteristic of heteroepitaxial systems.

During Year Three, we extended our examination of the ZnTe/GaSb system to layers deposited on GaSb with the (100) orientation – an orientation widely used for optoelectronic applications. The same abrupt change in x-ray FWHM values occurred for this system, but at a thickness in the range 350nm – 375nm, values 100nm less than those for the (211) orientation.

In response to suggestions from reviewers of a manuscript that we submitted with the (211) results, we brought additional experimental techniques to bear on this problem. We chose the techniques of cathodoluminescence, confocal photoluminescence, and transmission electron diffraction (TEM). These are imaging techniques. For other semiconductor materials, dark spots in luminescence images have been correlated with the emergence of dislocations at the surface of a sample. Certain dark lines in transmission electron micrographs are known to be associated with dislocations. Thus any dislocations in the ZnTe layer are directly imaged. Images from these techniques show dislocations forming in ZnTe layers as thin as 150nm.

We reconcile the formation of dislocations at 150nm and the advent of plastic deformation at 350nm by recourse to the concepts of dislocation generation and dislocation multiplication.

An extensive discussion is given in the manuscript entitled *Determination of Critical Thickness for Epitaxial ZnTe Layers Grown by Molecular Beam Epitaxy on (211)B and (100) GaSb Substrates*, a copy of which is appended to this report.

2. MBE of ZnSeTe

During Year Two, by following the peak position in x-ray diffraction spectra he had demonstrated that the alloy composition could be controlled by adjusting the ratio of Se to Te in the molecular beams incident on the substrate.

During year 3 we supplemented these x-ray results with images of the surface using atomic force microscopy, scanning electron microscopy, and micro photoluminescence. RMS roughness values of 1.1nm are higher than those for the binary ZnTe endpoint. However, for ZnSeTe layers that are closely lattice matched to the GaSb substrate, the density of dark spots in μ PL images is as low as $7 \times 10^4 \text{ cm}^{-2}$. These values are grounds for optimism that the GaSb/ZnSeTe composite system will be an excellent substrate for deposition of HgCdSe. By way of comparison, CdZnTe wafers that are the baseline substrate for HgCdTe epitaxy in the US IR industry have a dislocation density specification of mid 10^4 cm^{-2} .

An extensive discussion of these results is given in the manuscript entitled *Growth of Lattice-Matched ZnTeSe Alloys on (100) and (211)B GaSb* and in the presentations entitled *Comparison of Cathodoluminescence, Imaging Micro-Photoluminescence, and Confocal Photoluminescence Microscopy for Determination of Defect Densities in Semiconductors*. Copies of both are appended to this report.

3. MBE of HgCdSe

Prior to this ARO program, typical electron concentrations for both bulk and thin film samples of HgCdSe were between 10^{17} and 10^{18} cm^{-3} , with some samples containing concentrations as high as 10^{20} cm^{-3} . These high electron concentrations persisted at lower temperatures, showing little temperature variation below 100K. For IR pv diode detectors, target value for electronic background level is $5 \times 10^{14} \text{ cm}^{-3}$.

During Year Two of this program, we had achieved background electron concentrations of $1 \times 10^{17} \text{ cm}^{-3}$.

During Year Three, we carried out an intensive investigation of defects in these layers with an eye toward identifying those that are responsible for the high electron concentration. Composite GaSb/CdSeTe substrates with acceptable properties were not available at the beginning of Year Three. What was available was a Si/ZnTe composite that had been highly developed at Army Research Laboratory over a period of many years. This substrate was used for all HgCdSe work during Year Three. Growth rate, composition, and surface quality were evaluated for various MBE parameters. Two types of cell were used for Se evaporation – an effusion cell containing 5N purity material that produced a flux that consisted mainly of the species Se_6 , and a disassociation source containing 6N purity material that could be operated in temperature regimes that produced mainly Se_2 or mainly Se_6 species. For a given substrate temperature and Hg overpressure, the growth rate was controlled by the Se flux and the x -value was controlled by the Cd/Se flux ratio. Growths under Hg-deficient conditions produced “needle” and “diamond”- shaped defects at the layer surface. The optimal substrate temperature was found to be 90-110 °C for growths performed with the Se_6 flux from the effusion cell and a standard Hg flux of 2.5×10^{-4} Torr.

A time-honored technique for filling host-atom vacancies in MBE layers is to anneal the layers in an ambient of a host atom. For HgCdTe, for example, annealing layers in a

mercury ambient is found to fill Hg vacancies that remain after the MBE process. During Year Three, we carried out an extensive study of the effects of annealing HgCdSe layers. Positron annihilation spectroscopy measurements were carried out. These suggest the presence of p-type mercury vacancies in $\text{Hg}_{1-x}\text{Cd}_x\text{Se}$ samples both as grown and after annealing under an Se overpressure. Temperature-dependent Hall measurements of annealed samples suggest two donor energy levels: one in the band gap with ionization energy of ~ 40 meV that produces an electron concentration of $\sim 8 \times 10^{15} \text{ cm}^{-3}$ at 300K, and one in the conduction band with a concentration of $\sim 2 \times 10^{16} \text{ cm}^{-3}$. The former could originate from Se vacancies that act as donors, while the latter is now thought to correlate with impurities in the Se source material.

The lowest value measured for electron concentration was $8 \times 10^{15} \text{ cm}^{-3}$. Thus we have made a great stride toward the target value for IR detectors.

A prerequisite for achieving high responsivity values for pv diode IR detectors is a high minority carrier lifetime in the absorber layer. Near the end of Year Three, we carried out photoconductive decay measurements on HgCdSe layers to determine the lifetime. All samples were given an anneal for twenty-four hours. Lifetime was found to depend strongly on the species in the ambient during this anneal. If Se was present in the ambient, the photoconductive decay could be fit by a simple exponential function. The longest lifetime value obtained to date was on the order of 300 ns.

An more extensive account of these experiments and results can be found in the manuscript entitled *HgCdSe for Infrared Detection* and in the doctoral dissertation entitled *Development of $\text{Hg}_{1-x}\text{Cd}_x\text{Se}$ for 3rd Generation Focal Plane Arrays using Molecular Beam Epitaxy*, a copy of which is appended to this report.

4. Luminescence imaging technology

The RoA product for pv diodes is found to decrease as the density of defects such as dislocations increases in the absorber layer. This correlation is so strong that layers are screened prior to entry into device processing according to their dislocation density. Historically, chemical etchants have been developed that produce pits at the surface of a layer. Each pit corresponds to the emergence of a dislocation that threads through the layer to the surface. Unfortunately, no universal etchant has been developed. Each material and each crystallographic orientation of each material requires a unique etchant. No such etchant exists for HgCdSe or CdSeTe. It is important to develop an alternative to chemical etching for the determination of dislocation density.

In the interim report for Year Two of the ARO program, we described our application of the cathodoluminescence (CL) technique to determination of dislocation density. When CL is used for delicate semiconductor materials, care must be taken to maintain low current density for the stimulating electron beam to avoid damaging the sample surface. During Year Three, we extended our luminescence imaging technology by introducing two imaging techniques where luminescence is stimulated by a laser whose power density can be kept low enough to avoid damage. These are photoluminescence (PL) and confocal photoluminescence (cPL). We have utilized both techniques to acquire images

from ZnTe layers deposited on GaSb substrates and, by counting dark spots, have arrived at values of dislocation density for these layers.

An extensive account of the microscopy and the results can be found in the presentations entitled *The Use of Confocal Photoluminescence Microscopy for Determination of Defect Densities in Various II-VI Semiconductors, Comparison of Cathodoluminescence, Imaging Micro-Photoluminescence, and Confocal Photoluminescence Microscopy for Determination of Defect Densities in Semiconductors, and Comparison of Cathodoluminescence, and Imaging Micro-Photoluminescence and Confocal Photoluminescence Microscopy for Determination of Interfacial Defect Densities in Semiconductors*, copies of which is appended to this report. Where applicable, we have conclusively demonstrated that cPL is by far the best technique.

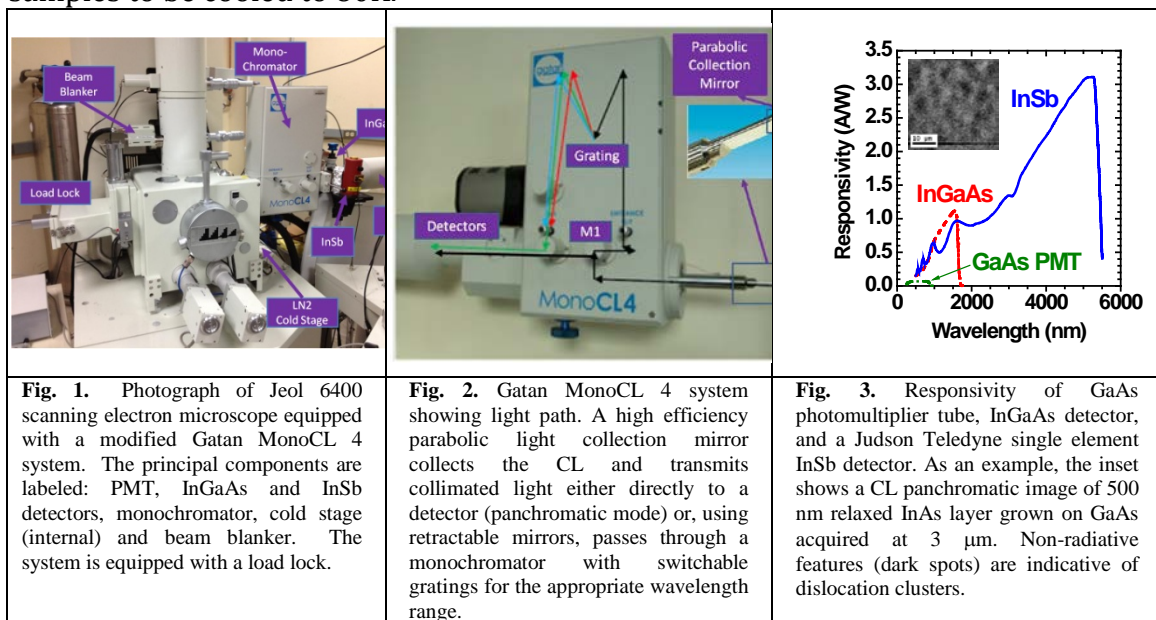
5. Investigate the technique of cathodoluminescence (CL) for various IR layers such as SLS layers

CL is non contact and non invasive. Luminescence generated by the electron beam in an SEM can be detected and images of luminescent intensity can be constructed by rastering the beam across a sample. Dislocations that intersect the surface strongly reduce the intensity of the intrinsic luminescence. Spectral distributions of CL intensity show peaks at energies corresponding to the band gap of the material. For wide bandgap materials such as CdTe and ZnTe, detectors and optical systems are readily available. For narrow bandgap materials such as InAs, GaSb/InAs SLS, HgCdTe, or HgCdSe, this is not the case. Only a small number of systems even extending to about 2 μm are known to be operational in the world.

The bandgap of LWIR material is 0.1 eV. Any CL apparatus for use with this material must have mirrors and a detector that are appropriate for wavelengths as long as 12 μm . Texas State University has designed and built an instrument for acquiring CL images and therefore dislocation density estimates for wafers and epilayers with bandgaps as low as 0.1 eV. We worked with Gatan, and have developed a unique IR CL system which is currently the only one of its kind in the world. It has the following features:

- High collection efficiency, precision diamond turned paraboloidal ir mirror. ~75% of all light is collected compared to 1-5% typical of CL and PL systems
- High transmission efficiency infrared optics for measurement out to 12 μm
- Visible (250-930 nm PMT), NIR (InGaAs 2.2 μm), MWIR (InSb) and LWIR (HgCdTe) detectors
- Direct optical coupling to chamber mounted monochromator
- Simple mirror switching allows light to transmit through monochromator, or bypass straight to the appropriate detector
- Fast beam blanking and detectors allows transient CL measurement with > 50 ns decay times, with the potential to allow direct mapping of lifetime across a sample.

CL is measured using a Jeol 6400 scanning electron microscope (Fig. 1) with a modified Gatan MonoCL 4 system (Fig. 2). This is the only known system that has been modified with IR optics compatible to 12 μm operation, enabling new metrology for III-V CMOS relevant metrics. For this program, NIR and MWIR detection were accomplished using a high sensitivity GaAs photomultiplier tube (250-950 nm), an InGaAs detector (2.2 μm), and a Judson Teledyne single element InSb detector operating at 80K (5.5 μm), respectively (Fig. 3). The MWIR signal was measured using a lock-in amplifier, with a Deben beam blanker modulating the electron beam incident on the sample. This allowed separation of the dc IR background from the ac CL signal while phase sensitive detection increased detectivity by 1000X. Both spectral and imaging (monochromatic or panchromatic) CL can be obtained from samples. The SEM was equipped with a cold stage allowing samples to be cooled to 80K.



The following figures consist of data collected with the CL system demonstrating its utility in the MWIR. We are actively working to push the detection out to the LWIR as one of the goals of this project.

Figure 4 illustrates the CL collected from a SLS mesa detector structure. The center consists of a metal contact, with the surrounding top layer nominally 3- μm cutoff while the base is nominally 5- μm . No signal was observed from the metal, as expected, and CL from the mesa to and base region were clearly observed. There was no evidence of non-radiative defects in this measurement, but two observations are pertinent. First, the darker region on the lower figure was not a measurement artifact, and the contrast became more pronounced with lower accelerating voltage (more near-surface collection). Second, we could not observe any CL in

the base region for accelerating voltages less than 10 keV, suggesting there is a significant “dead layer” in this unpassivated etched sample. Both observations suggest that the CL measurement is sensitive to surface recombination, with the former feature perhaps being related to a difference in surface etching. The latter indicated that CL will be useful in assessing the efficacy of surface passivation and its influence on quantum efficiency.

The dead layer in the etched samples reduced the CL signal and made data collection time intensive. We also looked at two so-called “PL Structures” where heterojunctions were used to reflect carriers from the surfaces and minimize surface recombination. The brighter CL allowed spectra data collection and many more images to be collected. Figure 5 and Figure 6 illustrate the spectral CL obtained from these samples, while Figures 7 and 8 illustrate non-radiative defects observed in the two samples. Figure 9 shows an image suggestive of a dark-line defect. Overall the defect density was found to be in the 10^5 cm^{-2} or lower range in the three samples investigated with isolated clusters of high density. Undoubtedly the defects and clusters observed would be deleterious for device operation. CL imaging will prove a valuable diagnostic tool for SLS-based FPA development.

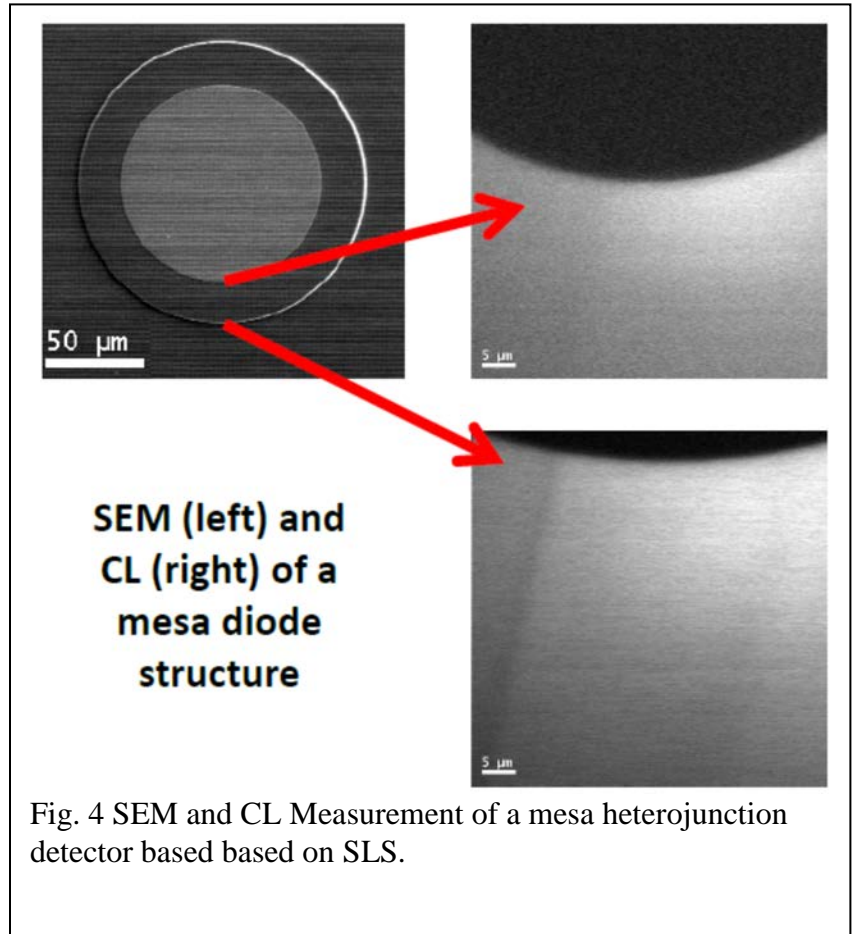


Fig. 4 SEM and CL Measurement of a mesa heterojunction detector based based on SLS.

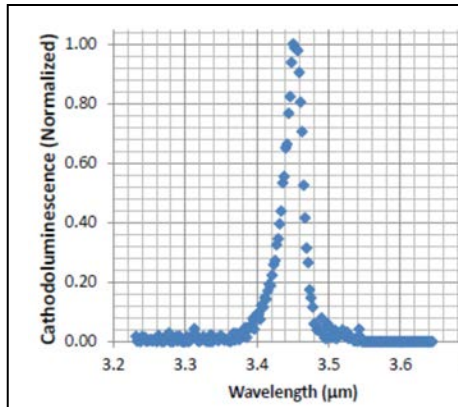


Fig. 5. Spectral CL collected from a nominal 3-μm PL structure.

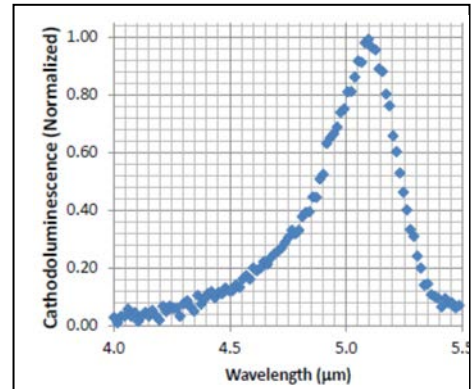


Fig. 6. Spectral CL collected from a nominal 5-μm PL structure.

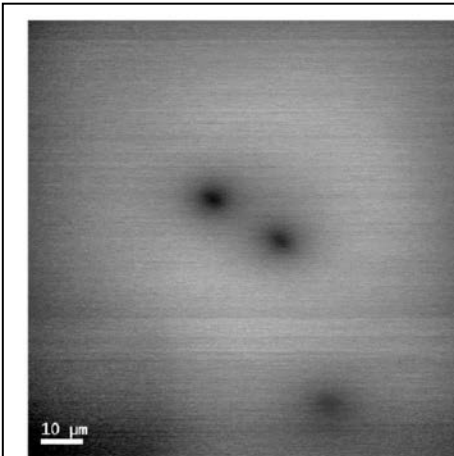


Fig. 7 CL image showing a typical defect distribution observed in a nominal 3-μm PL

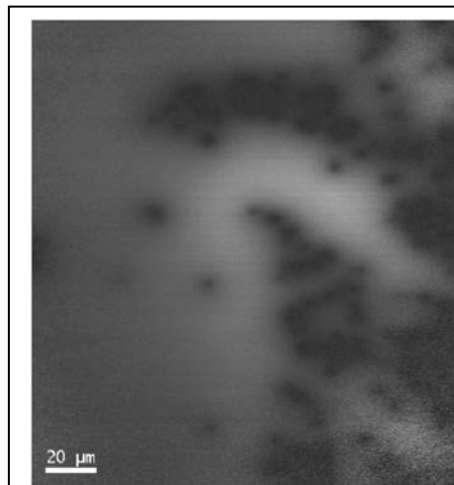


Fig. 8 CL image showing a defect cluster a nominal 5-μm PL structure.

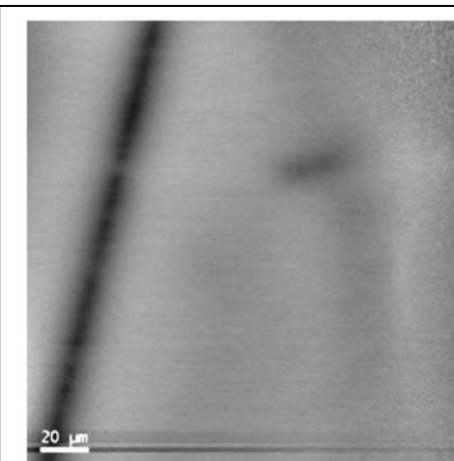


Fig. 9 CL image showing a defect feature in a nominal 3-μm PL structure reminiscent of a so-called “dark-line defect”.

